

FRACTOGRAPHY OF CRYOGENIC CHILL CASTED ASTM A 494 M GRADE NICKEL ALLOY METAL MATRIX COMPOSITES

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ABSTRACT

ASTM A 494 M grade Ni–Garnet composites containing four different weight percentages 3%, 6%, 9% and 12% of Garnet samples have been fabricated by using cryogenically cooled copper chill stir casting method. Effects of volume percent of Garnet particles on tensile strength, and fracture surfaces of Nickel Garnet composites have been investigated. The highest tensile strength was achieved in the specimen containing 9 Wt. % garnet produced with 25 mm chill thickness which shows an increase of 14% in comparison to the no chill cast reinforced alloy. Microscopic investigations of fracture surfaces revealed that fracture in a brittle manner with little or no necking happening. By increasing garnet content and chill thickness, the composites fracture goes in a more severely brittle manner. The fracture behavior of the composites was altered significantly by the presence of garnet particles and the crack propagation through the matrix and the reinforcing particle clusters resulted in the final fracture.

Key words: Nickel Alloy, Garnet, Cryogenic, Fractography, Metal matrix Composites.

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1. INTRODUCTION

Metal Matrix Composites (MMC's) are considered a group of advanced materials which represent low density, good tensile strength, high modulus of elasticity, low coefficient of thermal expansion, and good wear resistance. The production and use of composite materials is under intensive development because of the interesting physical and mechanical properties and also due to the possibility to manipulate them by means of the variation of the type and proportion of the reinforcement employed as well as the type of the metallic matrix. The particulate reinforced composites are gaining importance now a days, because of their low cost with advantages like isotropic properties and the possibility of secondary processing facilitating fabrication of components¹⁻³.

Composites containing reinforcement particles show increased modulus and wear endurance, but reduced tensile strength and high-cycle fatigue resistance⁴⁻⁵ by comparison with particle- reinforced composites. As for low cycle fatigue life, that of large-particle reinforced composites raises at higher strain amplitudes, but decreases at lower strain amplitudes⁶. However, investigations on the effect of reinforcement particle on low-cycle fatigue behavior of the composites are still limited⁷, especially the composite produced using cryogenic chills with varying sizes. Nickel-base super alloy that possesses excellent corrosion and oxidation resistance coupled with good tensile and creep properties. As a result, this alloy is extensively used in the structural application in the aerospace, petrochemical and also in automotive industries. Structural integrity assessments of such high strength structures require a comprehensive understanding of the fracture toughness characteristics for this super alloy. In addition, nickel alloy are selected for several high temperature applications which are highly loaded during service, so fracture control is a primary design consideration⁸⁻¹¹. The present study investigates the fractographic behavior of Ni-Garnet composites developed with varying percentage of reinforcement into a matrix material, at varying chill thickness. The present research is an attempt to test and analyze the mechanical properties of Nickel alloy reinforced with up to 3 - 12% by weight of garnet reinforcement particles produced by using the cryogenic chill and stir casting method. And also the evaluations of fracture toughness of ASTM A 494 M grade nickel alloy with varying weight fraction of particle reinforced Garnet MMCs developed by using cryogenically cooled chill stir casting technique are presented in this work¹²⁻¹⁷.

2. EXPERIMENTAL PROCEDURE

2.1. Material Selection

2.1.1. Matrix Material

The chemical composition of the selected “ASTM A 494 M grade nickel base alloy” matrix material is given in the Table 1.

Table 1 Chemical composition of matrix material ASTM A 494 M grade nickel base alloy. (Inconel-625)

Elements	% by wt.
Nickel	Balance
Chromium	20.0-23.0
Iron	5.0
Molybdenum	8.0-10.0
Niobium (plus Tantalum)	3.15-4.15
Carbon	0.10
Manganese	0.50
Silicon	0.50
Phosphorus	0.015
Sulfur	0.015
Aluminum	0.40
Titanium	0.40
Cobalt	1.0

2.1.2. Reinforcement

Reinforcement material selected was Garnet, a group of silicate minerals, which is one of the hardest naturally available ceramic material. The chemical composition of the selected garnet is given in the Table 2.

Table 2 Chemical composition of Almandine Garnet ($\text{Fe}_3\text{Al}_2\text{Si}_3\text{O}_{12}$)

Elements	% by wt.
Aluminium Oxide	19.0
Iron Oxide	34.10
Calcium Oxide	3.0
Magnesium Oxide	4.51
Titanium Oxide	2.80
Manganese Oxide	0.58
Silica	35.90

2.2. Metal Matrix Composite Preparation

A stir casting process was used to fabricate the nickel base matrix alloy fused with Garnet, having reinforcement particles varying from 3wt. % to 12wt. % in steps of 3wt% for the preparation of metal matrix composites. The matrix alloy was melted in a casting furnace at around 1350°C shown in Figure 1. At the same time the garnet particulate was preheated in another furnace set at 600°C for approximately 2 hour to remove surface impurities and assist in the adsorption of gases. Then the preheated 3 wt. % of garnet particulates, were introduced evenly into the molten metal alloy. This process was repeated for 6, 9 and 12 wt. % reinforcement. Simultaneously, the

molten metal was well agitated by means of a manual mixing using graphite stirrer, which was carried out for about 5 min.

The moulds were prepared using silica sand with 5% bentonite as binder and 5% moisture according to American Foundry Society (AFS) standards, and were dried in an air furnace. The moulds prepared were rectangular bar shaped ingots of dimensions $150 \times 40 \times 25$ mm as per ASTM standards. A chill block was placed adjacent to one end of the mould. The arrangement of sand moulds and chill blocks is shown in Figure 2. Also the arrangements were made in chill blocks to circulate the liquid nitrogen in and out for cryogenic effect. The chill blocks placed were made up of copper of thickness 10mm, brazed with hollow MS blocks of size $150 \times 35 \times 40$ mm are shown in the Figure 3. The molten material at 1350°C was next poured into the sand mold. Liquid nitrogen was introduced into hollow steel block before and after pouring of the molten mixture for cryogenic effect. The above same procedure was repeated for chill thickness of 20 and 25 mm. The same type of sand mould was also used to cast a specimen without chilling effect.



Figure 1 Casting furnace



Figure 2 Sand mould with Copper end chill and arrangements for passing liquid nitrogen



Figure 3 Brazed copper chill with steel hollow block for passing liquid nitrogen

2.3. Tensile Test

To study the tensile behaviour and to determine the ultimate tensile strength of the matrix composites, specimens were prepared and tested as per ASTM E8M¹⁸ standard as shown in Figure 4. The specimens were machined using wire cutting. Tensile test were performed using Universal Testing Machine model: TUE-C –400.



Figure 4 Tensile test specimens

2.4 Fractographic Test

In order to investigate the fracture surfaces of the composite specimens, topographic observations were carried out using a Zeiss make model SEM. The fractured specimens of the tensile tests were considered shown in Figure 5.



(a)



(b)



(c)



(d)



(e)



(f)



(g)



(h)

Figure 5: Fractography test specimens (a) 3 wt. % Garnet without chill, (b) 3 wt. % Garnet with 25mm thickness chill, (c) 6 wt. % Garnet without chill, (d) 6 wt. % Garnet with 25mm thickness chill, (e) 9 wt. % Garnet without chill, (f) 9 wt. % Garnet with 25mm thickness chill, (g) 12 wt. % Garnet without chill, (h) 12 wt. % Garnet with 25mm thickness chill.

3. RESULTS AND DISCUSSION

3.4. Tensile Strength

Table 3 Mechanical properties of matrix material

Matrix material	UTS (MPa)	BHN	Yield Strength	% elongation
ASTM A 494 M	485	163	275	25

Table 4 UTS in N/mm² of cryo-chilled reinforced metal matrix cast using copper chills of varying thickness.

Chill thickness in mm	Nickel alloy			
	3 wt.% of Garnet	6 wt.% of Garnet	9 wt.% of Garnet	12 wt.% of Garnet
10	493	514	555	542
20	498	518	575	560
25	520	553	635	598
No chill	490	499	542	530

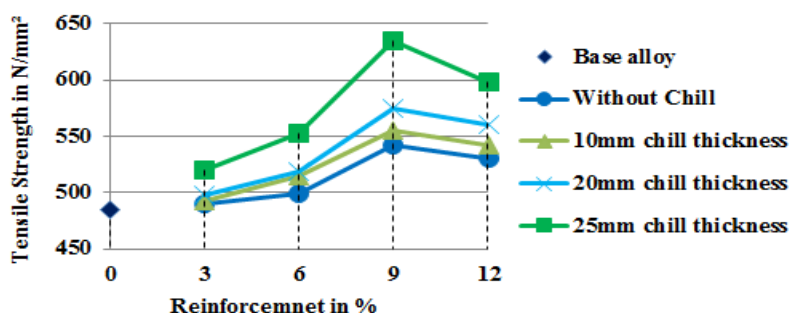
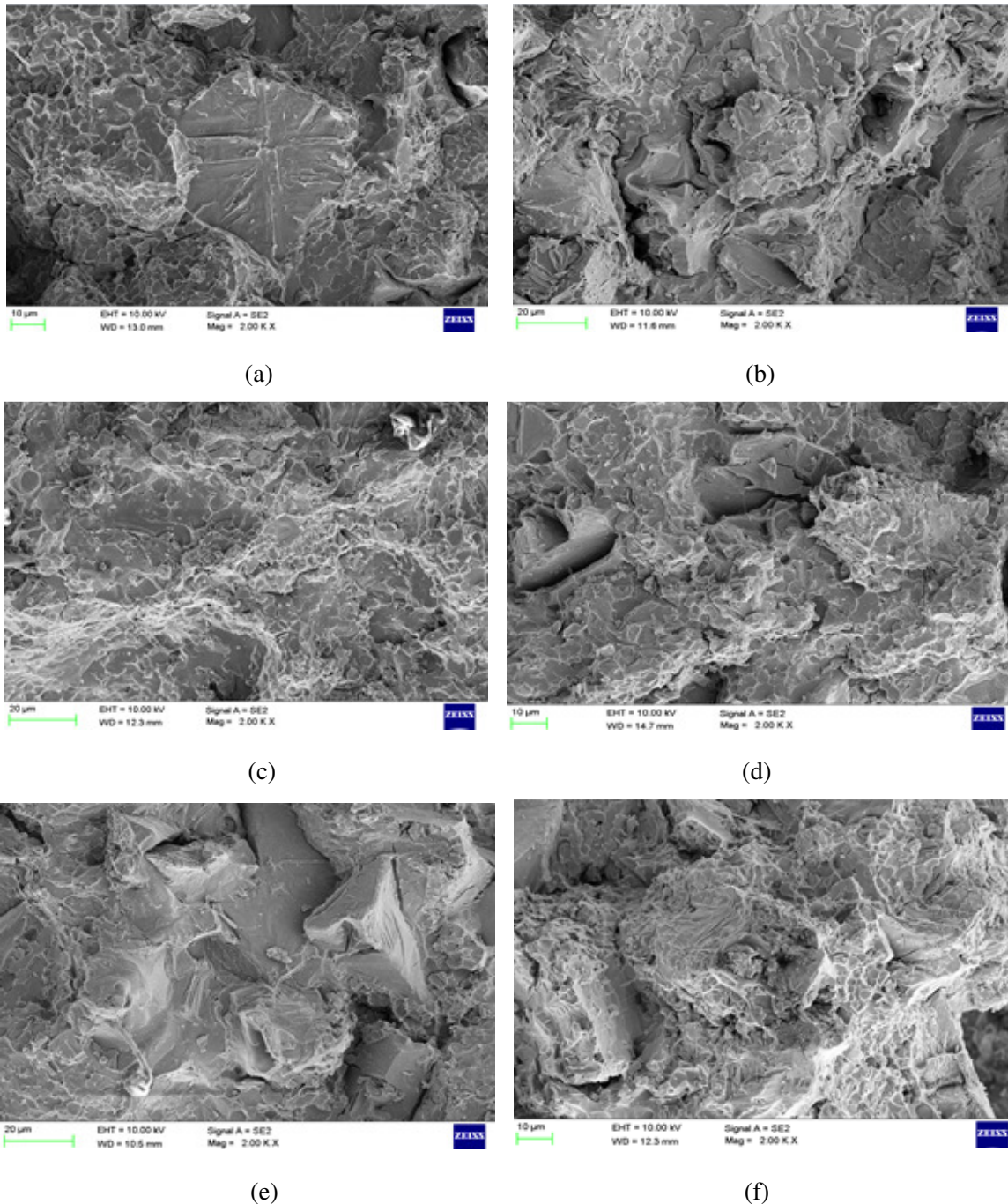


Figure 6 Tensile strength of Nickel based composite with varying chill thickness and % Garnet reinforcement

The tensile testing of the developed composite result in the Table 4 shows the ultimate tensile strength (UTS) measured by considering the specimen from the chill end for Nickel matrix/reinforced composites cast of different thickness. It is observed that the tensile strength of no chill cast composite are lower than that of the remaining chill cast composite with varying chill thickness. As the chill thickness increases, UTS also increases confirming that the volumetric heat capacity (VHC) of the copper chill along with liquid nitrogen significantly enhances the grain structure. As the reinforcement content is increased; the tensile strength is also increases. The increase in tensile strength is due to the presence and uniform distribution of garnet reinforcements which is having inferred high strength, and grain structure obtained from the cryogenic chilling. UTS of different cast composites with varying reinforcement weight percentage and chill thickness is shown in Figure 6. The result shows that tensile strength is increasing up to 9 wt. % Garnet content and then start gradually decreasing for 12 wt. %, where the trend is reverse.

3.3. Fracture surface

Figure 7 show the SEM micrographs of the fracture surfaces of the specimens containing 3, 6, 9 and 12 wt. % Garnet reinforced Nickel MMC produced using 25mm chill thickness and without chill. These images are taken by secondary electrons in order to study the fracture mechanisms of Ni- Garnet composites.



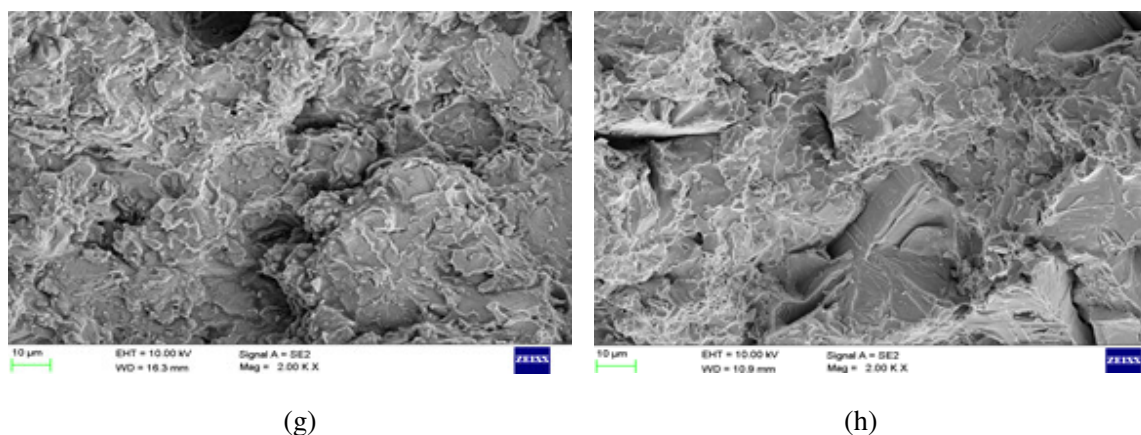


Figure 7 SEM images of the fracture surfaces of the composite Nickel - Garnet specimens containing (a) 3 wt.% Garnet without chill, (b) 3 wt.% Garnet with 25mm thickness chill, (c) 6 wt.% Garnet without chill, (d) 6 wt.% Garnet with 25mm thickness chill, (e) 9 wt.% Garnet without chill, (f) 9 wt.% Garnet with 25mm thickness chill, (g) 12 wt.% Garnet without chill, (h) 12 wt.% Garnet with 25mm thickness chill.

The fracture observed in the composites depends on a variety of factors, fracture of the reinforcing particles¹⁹⁻²⁰; partial de-bonding of particle-matrix interface and nucleation of voids²¹; growth of the voids and initiation of cracks in the matrix²².

In brittle fracture, reinforcing particles are not observed in surfaces, while eutectic phases are mostly present. Crack initiation takes place from these areas and the fracture surfaces of the composite contains almost no fractured garnet particles, but instead, display evidence of the eutectic and particle pullout. These eutectic phases appear brighter in the SEM images than other areas. But in ductile fracture, the reinforcing particles start to crack first and then crack growth takes place from these particles. This is the reason for observing fractured pieces of reinforcing particles in topographic images. Figure 7 shows the SEM images of typical fractured surfaces of Ni-garnet composite obtained from tensile tests. SEM examination of the fractured surface of the reinforced material with chill contained only smaller dimples than the fractured surface of the reinforced material without chill. In 3wt. % reinforced composites with and without chill composite specimens showed brittle fracture failure as shown in fig 7(a, b).

Ductility failure is observed in the fracture surface of 6 wt. % Garnet reinforced without chill composites and the same trend is also seen in 6 wt. % Garnet reinforced with chill composites shown in Fig. 7(c, d). Only very few fracture can be seen as fractured and there is also an evidence of ductile failure in the matrix. The failure showed few fracture split longitudinally and transversally as shown in Fig 7(e, f). The failure of fracture in the composite shown in fig (g,h) may be attributed to the increase in stress on the specimen. As the load on the fracture increases, it induces strain in the particles and the most heavily loaded fracture failure.

4. CONCLUSIONS

1. Mechanical property characterization of composite cast using 10mm, 15mm and 25mm thick copper chill block containing 3 to 12 wt.% reinforcement revealed that the presence of garnet particulates in nickel matrix has significantly improved tensile property by 14% (in case of 25 mm copper end chill thickness).
2. By increasing the Garnet content up to 9wt%.in the specimens, the tensile strength also increases. This is due to the accumulation of dislocation behind garnet particles which act as barriers on the movement of dislocations.
3. Increasing the Garnet content in the specimens above 9 % wt. Has almost no significant effect on the tensile strength.

4. According to the topographic observations, the fractures of Ni-garnet composites are brittle, since the matrix fracture is dominant and almost no fractured garnet particles are observed.
5. Fracture toughness also increases from 3 wt. % to 12 wt. % of adding the reinforcement.
6. Fractography analysis revealed that fracture behavior of Nickel matrix alloy was changed from ductile mode of fracture to cleavage mode because of the presence of garnet particles.

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